Progress on a Microgravity Dilution Refrigerator

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We have developed a shallow, single-cycle, helium dilution refrigerator that contains rather coarse metal sponge to study the ability to control the location of the liquid helium for microgravity applications. We have tested the refrigerator on the ground while tilting to put the mixing chamber either somewhat above or somewhat below the still. We calculated that the system could be tilted between 5 and 10 degrees in either direction without interrupting the cooling. The initial test of this refrigerator gave cooling to below 0.060 K and operation for tilts of ± 16 degrees. The insights gained from this refrigerator allow the design of a continuously operating version.

Keywords: dilution (E); ^{3}He-^{4}He mixtures (B); space cryogenics (F)

Introduction

A dilution refrigerator (DR) is the most common precooling stage for adiabatic demagnetization experiments that can reach temperatures below 0.001K. Such temperatures are needed to study the many unique properties of superfluid ^{3}He. The DR can provide cooling at 40-50 mK for long periods while the heat of magnetization is being removed from the demagnetization stage. In order to make the advantages of the DR available to researchers that need the microgravity of space for their experiments, we are developing a continuously operating DR that will function in microgravity. Combined with an adiabatic demagnetization stage, this will extend the temperatures that can be reached in microgravity to 0.001 K, where a number of experiments on superfluid ^{3}He have been suggested.

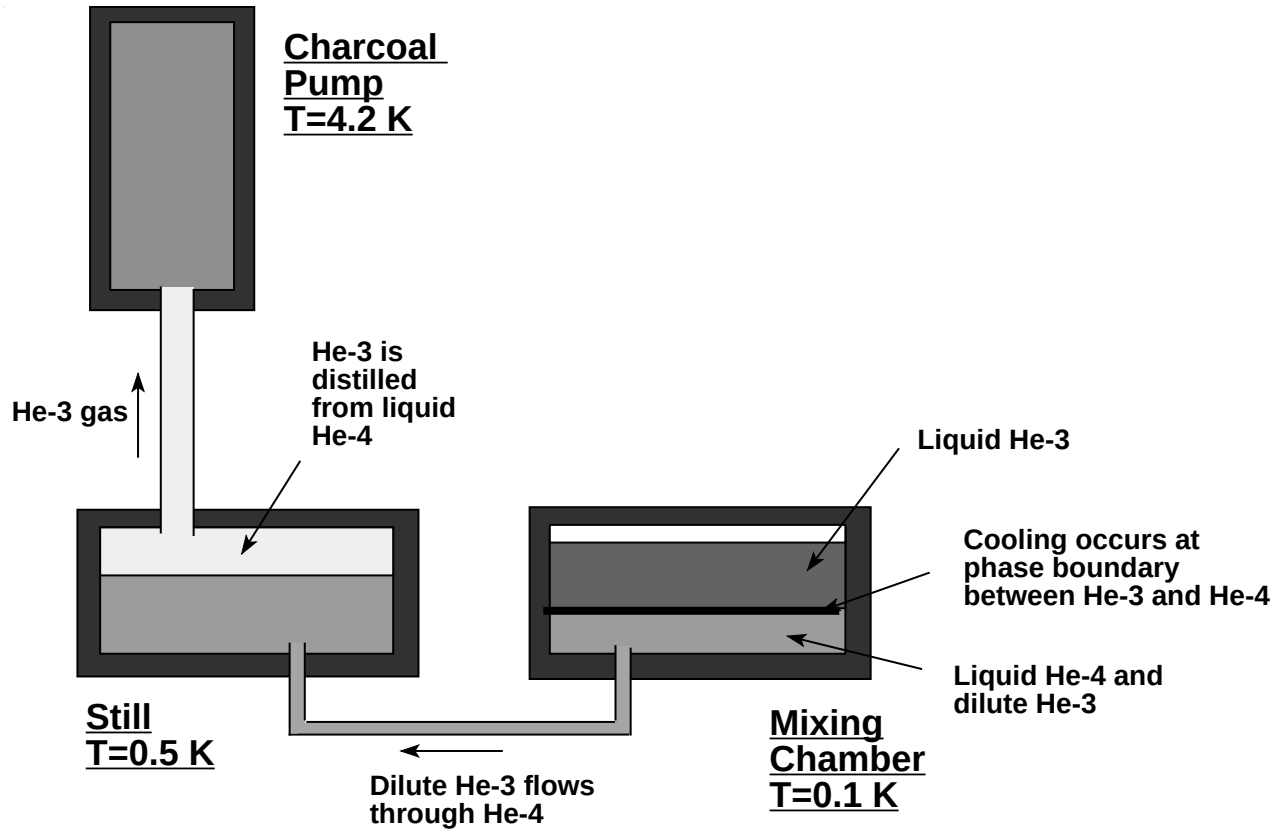

Fig. 1. Schematic of the operation of a single-cycle, sorption-pumped dilution refrigerator.

Figure 1 shows how a single-cycle, sorption-pumped dilution refrigerator[1] works. The lowest temperatures occur in the mixing chamber where there is a phase boundary between liquid ^{3}He and liquid ^{4}He. Cooling is produced when ^{3}He crosses this boundary into the ^{4}He. From the mixing chamber this dilute ^{3}He flows through the ^{4}He to a higher temperature chamber, the still, where it is fractionally distilled from the ^{4}He. The ^{3}He gas evaporating from the still is collected by the charcoal pump. The cooling cycle ends when all the ^{3}He is in the charcoal pump. Because the refrigerator uses adsorption onto charcoal for its pumping, all operations can be controlled by heaters and, as a consequence, there are no moving parts in the refrigerator and the helium gas never warms to room temperature during operation.

Modification for Microgravity

On the ground the operation of a DR depends on gravity to keep the liquid ^{3}He and ^{4}He in their correct chambers (the charcoal pump contains no liquid and is gravity independent). Within the DR there are two liquid-vapor interfaces and one liquid-liquid interface. All of these interfaces must be

stably located in the absence of gravitational forces in a way that allows the free flow of the evaporated gasses and of the ^{3}He within the liquid phases of the refrigerator.

Previous experiments[2] have shown that capillary forces in a matrix of fine pores can successfully contain liquid ^{3}He in microgravity. We have extended this approach to ^{3}He-^{4}He mixtures in a ground-based demonstration. The modifications we have made involve filling the liquid chambers of the dilution refrigerator with a sintered, porous metal matrix that confines the liquids to their correct positions by capillary forces. A critical aspect of this is the need to prevent the phase boundary between the liquid ^{4}He and liquid ^{3}He from leaving the mixing chamber. This is greatly complicated by the fact that the interfacial tension between these two phases is exceedingly small[3], only 2.0×10^{-5} N/m, compared with 1.5×10^{-4} N/m for the ^{3}He liquid-vapor surface tension and 3.5×10^{-4} N/m for the ^{4}He liquid-vapor surface tension. However, if the pores outside the region of the ^{3}He are small enough, and if the osmotic pressure trying to push the ^{3}He into these pores is not too large, the liquid ^{3}He will be prevented from entering the small pores containing the ^{4}He by the interfacial tension; the ^{3}He will stay in the mixing chamber where it is needed.

Shallow Dilution Refrigerator

In previous attempts to control the liquids in the DR by means of very fine pores we found that the small pores needed to control large heights of liquid on the ground are too small to allow sufficient liquid flow for effective cooling. For this reason we have developed a shallow version of the DR where the heights of the chambers are only 0.5 cm and the still and mixing chamber are next to each other instead of the still being above the mixing chamber.

Figure 2 shows the design of the shallow chambers used. Both the still and the mixing chamber include regions of larger pores and smaller pores. The large-pore regions act as reservoirs for the liquid; such reservoirs can provide liquid to the regions of smaller pores and prevent the small-pore regions from becoming empty too soon. If the pores of the connecting line or the junction region where the connecting line meets either chamber becomes empty of liquid, the circulation of ^{3}He will stop. The pore sizes of the various parts were chosen to allow the refrigerator to make maximum use of the ^{3}He available while making sure the liquids would be properly confined to the correct chambers under adverse accelerations. In the case of ground testing, adverse accelerations are provided

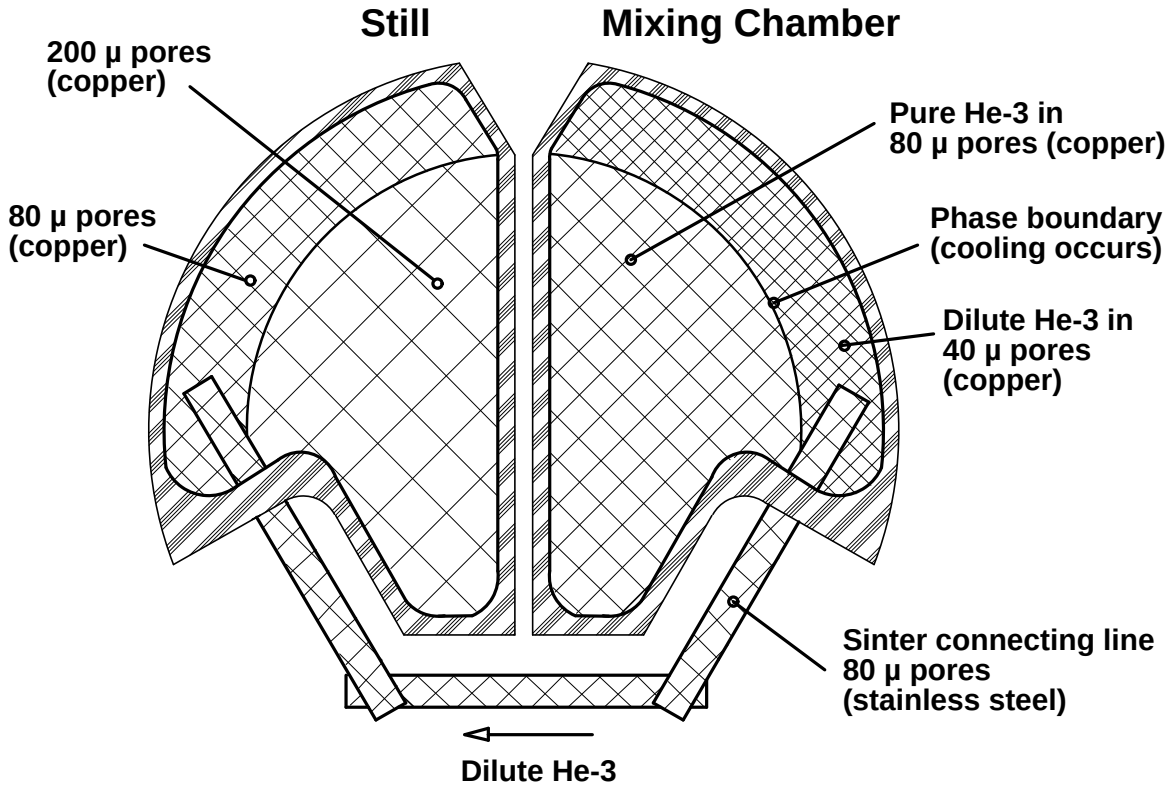

Fig. 2. Arrangement of different size sinters for confining liquid helium in a shallow still and mixing chamber.

by tilting the system so that the mixing chamber is somewhat above the still or somewhat below the still. Previous modeling of the system predicted that the refrigerator could tolerate tilts of 5-10 degrees in either direction.

Experimental Results

DR cooling: The most important result of our tests of this DR is that it cooled very well with the porous material in all chambers including the connecting line. Figure 3 shows a cool-down with the system tilted at –10° (mixing chamber lower than the still). Once the still begins to cool at a time of 14:45 the mixing chamber quickly starts cooling also. With 400 µW of heat applied to the still and no heat applied to the mixing chamber, the mixing chamber reaches 60 mK in about an hour. This result is significant because it means the porous material in the liquid chambers is not seriously impeding the flow of ^{3}He from the mixing chamber to the still. If the mixing chamber is heated to hold it at 100 mK while heating the still with 200 µW, then a cooling power of ~5 µW for the mixing chamber is obtained.

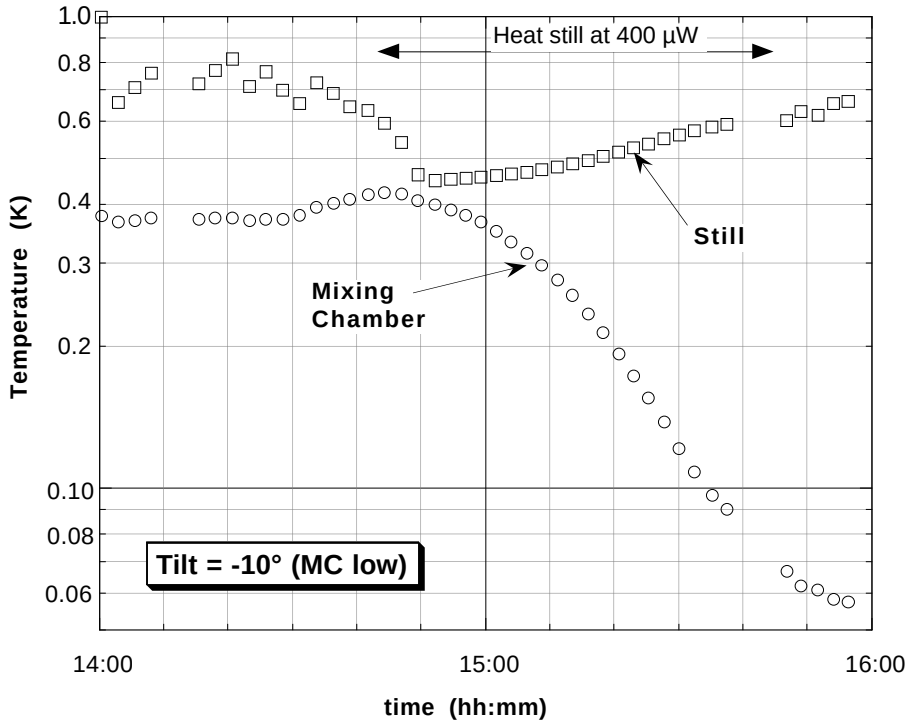

Fig. 3. Cooldown of DR to below 60 mK.

Tilting the DR: The operation of the refrigerator was studied at tilt angles from –20° (mixing chamber lower than the still) to +16° (mixing chamber higher than the still). In spite of our expectations that large tilts would prevent the operation of the DR, we observed cooling at all tilts that we tried (tilts beyond ±20° were not investigated because the dewar would require special support to prevent it from falling over). We did observe a strong dependence of the running time on the tilt angle, however. When tilted at +16° (MC high) (see Fig. 4), the still would draw ^{3}He from the mixing chamber for 145 minutes while being heated at 200 μW before the connection to the mixing chamber was lost. When tilted at –16° (MC low) (see Fig. 5), the still would draw ^{3}He from the mixing chamber for only 92 minutes at 200 μW before the connection was broken. Figure 5 also shows an unusual behavior that occurred at all negative tilt angles; after the pumping is begun on the still, a long period of time, 1 to 1.5 hours, would elapse with little or no ^{3}He coming into the still to allow any significant, steady cooling of the still. After this the still would cool normally and the mixing chamber would then start to cool down shortly thereafter. An explanation for this will be presented below.

Modeling results

In designing this shallow DR, a computer model was developed to predict the behavior of the

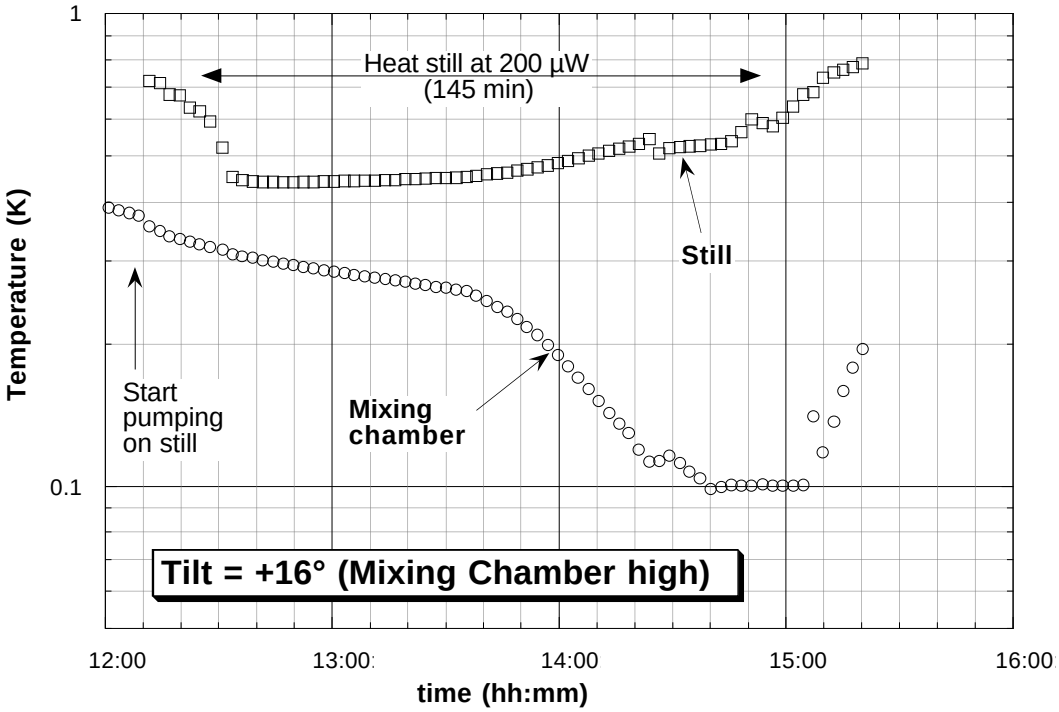

Fig. 4. Temperatures of still and mixing chamber during cooldown when tilted to +16° (mixing chamber high).

liquid in the still and mixing chamber in response to the hydrostatic heads and surface tension forces acting on the liquids in the various regions of the refrigerator. For any tilt angle the model calculates the net pressure on any element of liquid in the system and then moves small drops of liquid from chamber to chamber until the pressures balance in all chambers; the model assumes that ^{4}He can

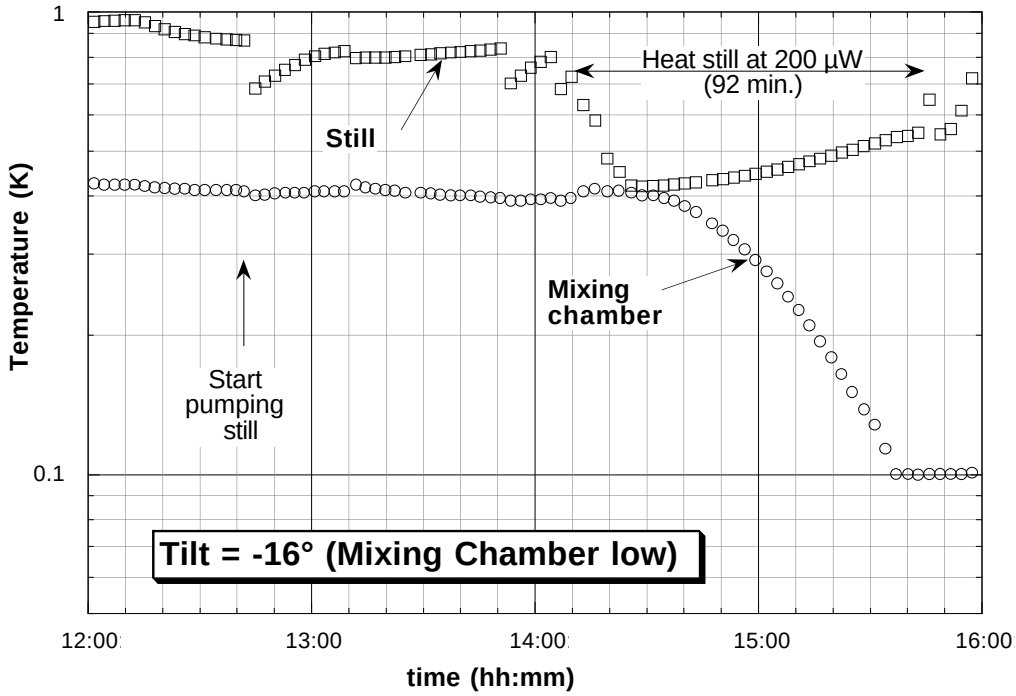

Fig. 5. Temperatures of still and mixing chamber during cooldown when tilted to –16° (mixing chamber low).

flow wherever it is needed so that the pressures in each region, when extrapolated to a common point, are equal. Without the influence of capillary forces in the porous materials in the chambers, the liquid ^{4}He would have the same level in all chambers. Capillary forces, however, can completely alter the distribution of liquid between regions.

This model was also used to interpret the results of the experimental tests. As the model was originally set up, however, there was no combination of reasonable pore sizes or initial liquid distributions that could explain the experimental results. It was not until we realized that the experiment was showing behavior not included in the model that we were able to explain what was happening. This behavior is the phenomenon that we call 'sticking', where liquid in the mixing chamber refuses to flow out when the mixing chamber is tilted up. This happens when the mixing chamber has been completely filled with liquid at some positive pressure and then cooled down. There is no void in the chamber then and liquid cannot flow out, even at large negative pressures, unless a bubble can be formed. At low temperatures it requires very large negative pressures to nucleate a bubble; we never achieve large enough negative pressures in our system.

When we modified the model to include the possibility of 'sticking', we were able to explain all the major features of our experimental data. Figure 6 shows the model prediction for the liquid

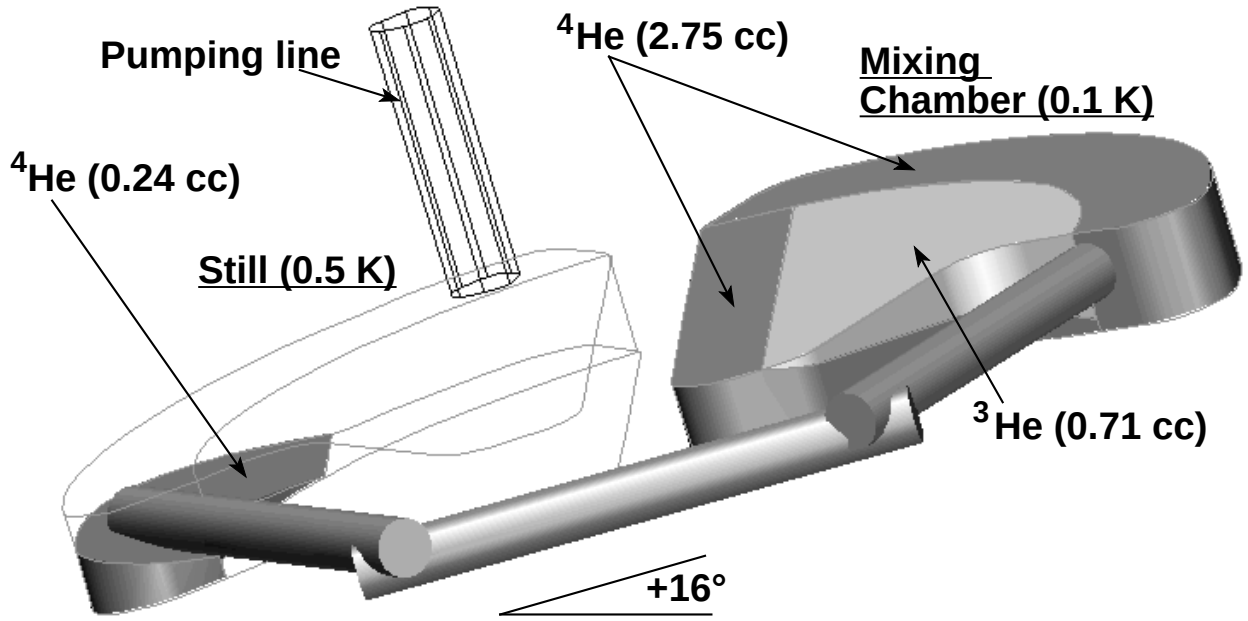

Fig. 6. Model prediction of liquid distributions in DR when tilted to +16°.

distribution in the DR for a tilt angle of +16° (MC high); here the liquid connection between the two chambers has just been broken because the still (and the end of the connecting line itself) has just gone empty where the connecting line joins it. Up until this time ^{3}He has been flowing through the ^{4}He from the mixing chamber reservoir, through the mixing chamber into the connecting line and into the still where it is distilled away. At the same time, ^{4}He has been flowing from the still into the mixing chamber to replace the ^{3}He since no void can form in the mixing chamber and its total volume of liquid must stay constant. It is primarily this flow of ^{4}He into the mixing chamber that depletes the still and causes the liquid connection between chambers to be broken. In this case, when the flow of ^{3}He stops there is relatively little of the original amount of ^{3}He left in the mixing chamber.

Figure 7 shows the results of the model calculation for a tilt angle of –16° (MC low). Again the mixing chamber is full when the liquid level in the still drops below the end of the connecting line and the liquid connection is broken In this case 'sticking' need not be invoked; the mixing chamber is full simply because capillary forces in the still cannot prevent the liquid from running into the

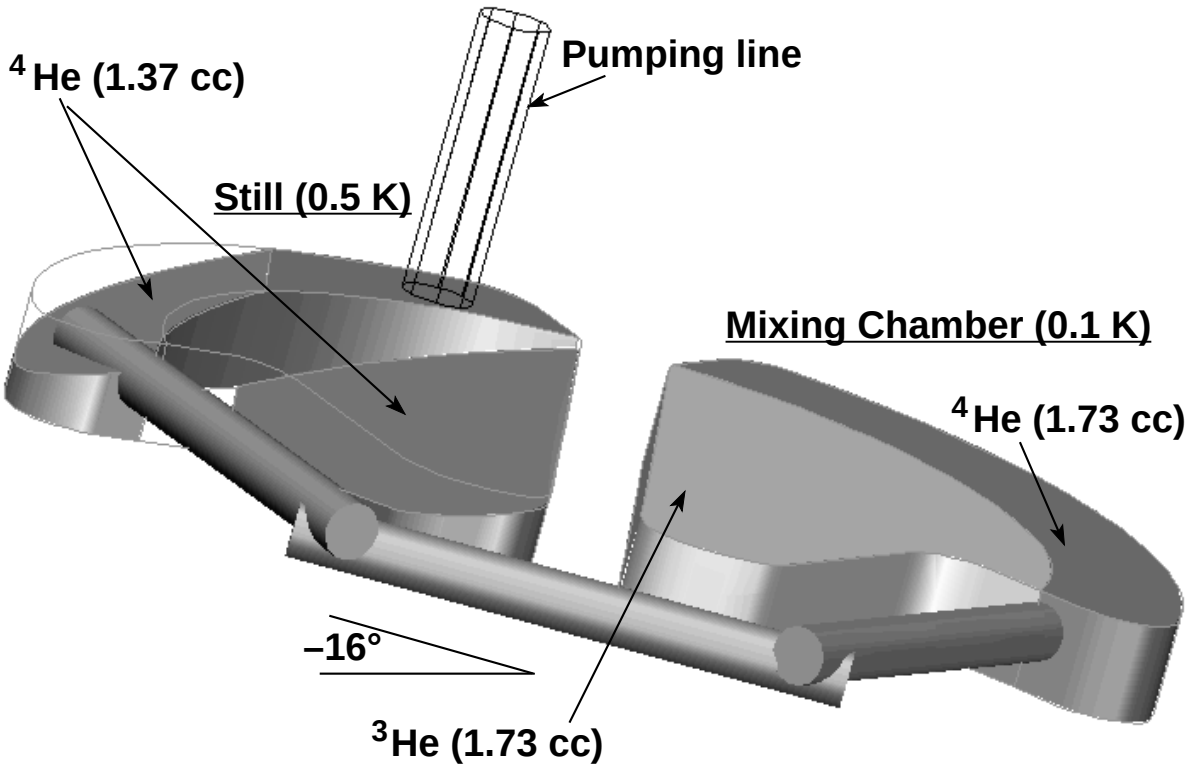

Fig. 7. Model prediction of liquid distributions in DR when tilted to –16°.

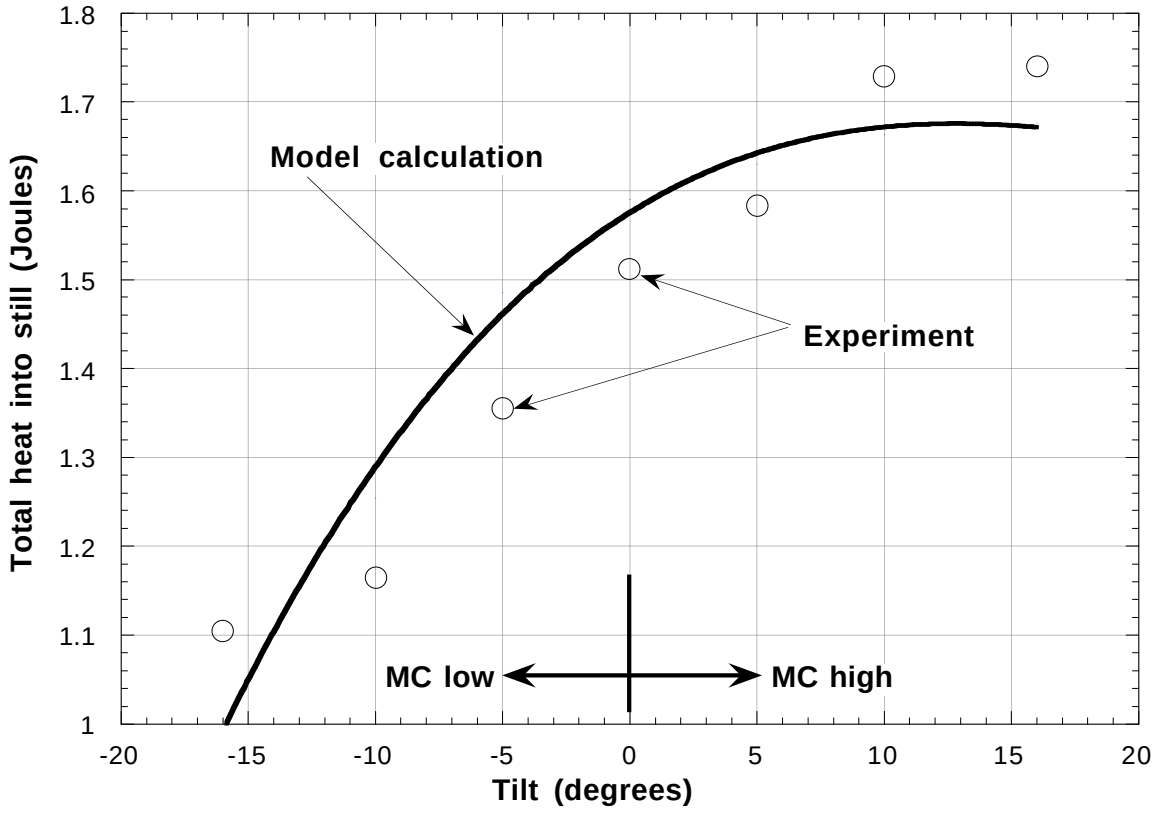

Fig. 8. Comparison of experimental results and model prediction for total heat that can be applied to the still to distill the available ^{3}He vs. tilt angle of the system.

mixing chamber at this large angle. Note that when the liquid connection is lost at this tilt angle, there is much more ^{3}He still remaining in the mixing chamber; this is why the running time at this angle is relatively short.

In using the model to describe the experimental data, it was necessary to somewhat modify the pore sizes given in Fig. 2. The best fit to the experiments was obtained by using a pore size of 95 μ instead of the nominal 80 μ size for the still, the connecting line and the mixing chamber reservoir; and a pore size of 150 μ was used instead of 200 μ for the still reservoir. These values are within the uncertainties of the pore sizes that we measured by using a bubble test in methanol at room temperature.

Figure 8 shows a comparison of the experimental results with the model calculation. The heat that can be applied to the cold still is a direct measure of the amount of ^{3}He that is flowing from the mixing chamber. The experimental points for total heat into the still come from the measured time during which 200 μW of heat could be applied, and the model calculation values are based on an

initial amount of ^{3}He in the mixing chamber of 3.25 cm^3, and a value of 24.4 J/mole for the latent heat of the ^{3}He distilled from the still. The agreement between experiment and the model gives convincing proof that the porous material in the still is behaving as predicted.

Because of the 'sticking' problem in the mixing chamber at positive angles, the experiment did not determine how well the porous material in the mixing chamber worked. This problem also prevented any test of the ability of the capillary forces to prevent the pure ^{3}He from flowing out of the mixing chamber reservoir. In the way the experiments were carried out, there was never any pressure on the ^{3}He in the reservoir trying to push it into the adjacent pores. This 'sticking' problem can be overcome by applying a pulse of heat to a small heater in the liquid in the mixing chamber to nucleate a bubble. This will allow the liquid to adjust its level in equilibrium with the hydrostatic pressures and capillary forces in the system. It is under these conditions that pressures trying to push the pure ^{3}He out of the mixing chamber reservoir can arise.

The behavior shown in Fig. 5, , where there is a long delay between the time pumping on the still is started and the time that significant cooling of the still begins, can also be explained by the model. We believe this problem is due to small amounts of pure ^{3}He that start in the small-pore mixing chamber region (outside the reservoir where they would be confined by capillary forces) flowing from the mixing chamber into the connecting line. The much lower surface tension of the ^{3}He causes the connecting line to drain empty at the position of the still, breaking the liquid connection to the mixing chamber. This situation lasts until the ^{3}He in the connecting line is depleted by diffusing back into the mixing chamber or by slowly distilling into the still through the liquid-depleted gap in the connecting line. Then ^{4}He then re-establishes the liquid connection because of its higher surface tension and some dilute ^{3}He can again flow to the still through the ^{4}He until another small amount of pure ^{3}He enters the connecting line. We have seen this process occur 5-10 times before a steady flow of dilute ^{3}He establishes itself.

Conclusions

The tests of this single-cycle, shallow DR have demonstrated that the porous material used does not seriously impede the flow of ^{3}He; proper operation of the dilution process is seen and cooling to

below 60 mK is obtained. A cooling power at the mixing chamber of 5μW is obtained when 200 μW is applied to the still. When allowance is made for the liquid 'sticking' in the mixing chamber, even at large tilt angles, our model is able to describe all the major behavior of the DR. This confirms our understanding of the effect of capillary forces on the liquids in the DR and gives us confidence in our ability to design systems to control these liquids in microgravity. We can now finalize our design [4] of a continuously operating dilution refrigerator.

REFERENCES

1. Roach, P. R. and Gray, K. E., "Low-Cost, Compact Dilution Refrigerator: Operation from 200 to 20 mK", Advances in Cryogenic Engineering, vol. 33, Plenum Press, New York (1988), pp. 707-712. (U.S. Patent #4,770,006)

2. Duband, L., Alsop, D., Lange, A.E., and Kittel, P., "^{3}He Refrigerators for Space", Advances in Cryogenic Engineering., vol. 35B, Plenum Press, New York (1990) p. 1447.

3. Guo, H. M., Edwards, D. O., Sarwinski, R. E., and Tough, J. T., "Fermi-Liquid Behavior of ^{3}He Adsorbed on Liquid Helium", Phys. Rev. Lett., vol. 27(19) (1971) pp. 1259-1263.

4. Roach, P. R. and Helvensteijn, Ben P. M., "Development of a Dilution Refrigerator for Low-Temperature Microgravity Experiment"s, Cryocoolers 10, Ed. R. G. Ross, Jr., Kluwer Academic/ Plenum Publishers, New York (1999), p. 647.